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Catalytic application of sulfonic acid-functionalized titanacoated magnetic nanoparticles for the preparation of 1,8dioxodecahydroacridines and 2,4,6-triarylpyridines via anomeric-based oxidation

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Funding information Iran National Science Foundation (INSF), Grant/Award Number: 95831207; National Elites Foundation We have developed green, efficient and powerful protocols for the preparation of 2,4,6-triarylpyridines and 1,8-dioxodecahydroacridines in the presence of Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H as a sulfonic acid-functionalized titanacoated magnetic nanoparticle catalyst under mild and solvent-free reaction conditions. These protocols furnished the desired products in short reaction times with good to high yields (20-40 min and 80-86% in the case of the of 2,4,6-triarylpyridines; 15-90 min and 80-93% in case 1,8-dioxodecahydroacridines). The final step of the mechanistic route in the synthesis of 2,4,6-triarylpyridines proceeds via an anomeric-based oxidation. Also, the nanomagnetic core-shell catalyst can be recycled and reused in both cases of the scrutinized one-pot multicomponent reactions with high turnover number and turnover frequency.

KEYWORDS

 $1,8-dioxodecahydroacridines,\ 2,4,6-triarylpyridines,\ anomeric-based\ oxidation,\\ Fe_3O_4@TiO_2@O_2PO_2(CH_2)_2NHSO_3H,\ multicomponent\ reactions,\ solvent-free\ reactions$

1 | INTRODUCTION

In contemporary research programmes, catalysis has become one of the most persuasive areas and has found an important position in developing fields of organic synthesis.^[1] Nanosized catalysis is one of the most influential fields for the synthesis of novel catalytic systems. Due to their small diameters, nanoparticles provide many profits like high external surface area, superior activity and selectivity and high reaction rates. However, the aggregation and also isolation and recovery of nanosized catalytic systems are the main defects linked with these versatile species. Employing appropriate supporting agents is a suitable approach for avoiding aggregation.^[2,3] One of the best choices for improving the stability and recyclability of nanomaterials is their immobilization onto appropriate magnetic supports. Magnetic nanoparticles are very useful alternatives for support materials in green chemistry research. Some of the benefits of these support compounds are as follows: eco-friendly benign nature, superior thermal and mechanical stability, easy production and functionalization, low cost and toxicity, easy handling and treatment and unequalled paramagnetic nature. In addition to these merits, constructed heterogeneous nanomagnetic catalytic systems compared with homogeneous analogues show very good catalytic performance with high isolation, recovery and reuse capability.^[4–7]

Nitrogen-containing heterocyclic molecules such as pyridine derivatives are important in the natural word. Among the pyridine ring scaffolds, aryl-substituted ones like 2,4,6-triarylpyridines which are known as Kröhnke pyridine motifs are particularly fascinating.^[8] These interesting structures have a unique position in medicinal chemistry and have various pharmaceutical and biological activities. They can be employed as intermediates for the preparation of medicinally active compounds such as those with antimalarial, anticonvulsant and antitumour activities^[9] and furthermore in agrochemical active materials such as herbicides, insecticides, desiccants and surfactants.^[10] Also, these molecules have been applied for polymers,^[11] preparation of organometallic the polyimides,^[12], photoluminescent polymers^[13] and chemosensors,^[14] and in asymmetric catalysis^[15] and supramolecules.^[16] Due to the previously mentioned medicinal benefits and applications connected with 2.4.6-triarylpyridine derivatives, a variety of protocols have been reported for their synthesis.^[17-23]

Other important nitrogen-containing heterocyclic structures are acridines and 1,8-dioxodecahydroacridine scaffolds. These compounds have found diverse utilities in laser dyes and photoinitiators.^[24] Additionally, in medicinal chemistry as pharmaceutical materials they can also serve as antimalarial, anticancer, cytotoxic, antimicrobial, anticancer and antifungal agents and are recommended as calcium β-blockers.^[25] Furthermore, due to the intercalation versatility of some natural and synthesized acridine derivatives, they are striking DNA- and RNA-binding structures.^[26] Considering the diverse applications of acridine derivatives, their preparation is a continuing focal point for synthetic chemists and several catalytic procedures have been investigated for this goal.^[27,28] However, most of these reported protocols encounter some drawbacks including long reaction times, unsatisfactory yields, large amount of catalyst loading, deactivation of catalyst, use of toxic volatile organic solvents, unpleasant reaction conditions and tedious workup procedure on repeated use.

In the area of combinatorial chemistry, one-pot multicomponent reactions have been identified as influential methods in the toolbox of synthetic chemists. Multicomponent reactions, due to their full compliance with criteria set by the principles of green chemistry, have been widely applied in the design, construction and improvement of synthetic organic methodologies.^[29–37]

In the work reported in this paper, by considering the above-mentioned 2,4,6-triarylpyridine derivatives and 1,8-dioxodecahydroacridines as significant nitrogen-containing structures and in order to develop new methodologies for the synthesis of heterocyclic molecules^[38–45] and expand our new introduced technique of 'anomeric-based oxidation' for the oxidation of target molecules,^[46] we studied the catalytic applicability of $Fe_3O_4@TiO_2@O_2PO_2(CH_2)_2NHSO_3H$ as sulfonic acid-functionalized titana-coated magnetic nanoparticles for the preparation of 2,4,6-triarylpyridines and 1,8-dioxodecahydroacridines under mild and solvent-free conditions (Schemes 1 and 2).

2 | RESULTS AND DISCUSSION

The sulfonic acid-functionalized titana-coated magnetic nanoparticles, namely $Fe_3O_4@TiO_2@O_2PO_2(CH_2)_2NHSO_3H$, were prepared based on our previously reported protocol as presented in Scheme 3.^[46b]

In the first stage, in attempting to investigate the catalytic performance of $Fe_3O_4@TiO_2@O_2PO_2(CH_2)_2NHSO_3H$



SCHEME 1 Synthesis of 2,4,6triarylpyridines in the presence of Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H

R= H, 4-Cl, 2-Cl, 4-Br, 3,5-(F)₂, 2-NO₂, 3-NO₂, 4-CN, 3-OH-4-Me, 2-OMe, 4-OMe, 3-OEt-4-OH, 3,4-(OMe)₂, 3,4,5-(OMe)₃,4-Me, Terephetaldehyde, Thiophen-2-carbaldehyde



SCHEME 3 Synthetic pathway for the preparation of $Fe_3O_4@TiO_2@O_2PO_2(CH_2)_2NHSO_3H$

as a sulfonic acid-functionalized titana-coated magnetic nanoparticle catalyst and in order to find the optimal reaction conditions in terms of amount of nanomagnetic catalyst, solvent and temperature in the case of 2,4,6WILEY-Organometallic 3 of 11 Chemistry

triarylpyridines, the reaction of benzaldehyde, acetophenone and ammonium acetate was selected as a model reaction (Scheme 4). The experimental results obtained from screening of reaction parameters are provided in Table 1. The highest yield and shortest reaction time were achieved in the presence of 10 mg of Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H at 110 °C under solvent-free conditions (Table 1, entry 2). Also, as evidence for the final step of the suggested anomeric-based oxidation mechanism for aromatization of desired molecules, we investigated the optimal reaction conditions under nitrogen atmosphere. The results for the model reaction under air and nitrogen atmosphere are similar. Elevating the reaction temperature and increasing the load of catalyst did not exhibit further positive effect. Also, performing the model reaction in common laboratory solvents including water,



SCHEME 4 Model reaction for screening of reaction parameters

Entry	Solvent	Temperature (°C)	Load of catalyst (mg)	Time (min.)	Yield (%) ^b
1	-	100	10	20	65
2 ^c	-	110	10	20	80
3	_	120	10	20	80
4	-	110	13	25	78
5	-	110	7	25	65
6 ^d	-	110	10	60	40
7 ^e	-	110	10	60	40
8	-	110	-	60	40
9	H ₂ O	Reflux	10	45	40
10	C ₂ H ₅ OH	Reflux	10	45	35
11	CH ₃ CN	Reflux	10	60	30
12	EtOAc	Reflux	10	60	35
13	CH_2Cl_2	Reflux	10	60	Trace
14	<i>n</i> -Hexane	Reflux	10	60	Trace

TABLE 1 Optimizing of the reaction conditions for the synthesis of 2,4,6-triarylpyridine derivatives^a

^aReaction conditions: benzaldehyde (1 mmol, 0.106 g), acetophenone (2 mmol, 0.12 g) and ammonium acetate (5 mmol, 0.385 g), ^bIsolated yields.

^cThe achieved data for testing the model reaction under air and nitrogen atmosphere are similar.

^dThe reaction was performed using Fe₃O₄ nanoparticles as catalyst.

^eThe reaction was performed using TiO₂ nanoparticles as catalyst.

EtOH, CH₃CN, EtOAc, CH₂Cl₂ and *n*-hexane did not lead to satisfactory results compared to solvent-free conditions.

Subsequently, after determining the optimal reaction parameters, to study the efficacy and applicability of Fe_3O_4 @TiO_2@O_2PO_2(CH_2)_2NHSO_3H as a sulfonic acidfunctionalized titana-coated magnetic nanoparticle catalyst in the preparation of target molecules, various arylaldehydes were treated with acetophenone derivatives and ammonium acetate to furnish the corresponding 2,4,6-triarylpyridines with good yields and short reaction times. Table 2 presents the experimental data from this new protocol.

In another exploration in the light of promising results from the construction of 2,4,6-triarylpyridine derivatives, we investigated the catalytic applicability of Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H in the condensation reaction between aromatic aldehydes, dimedone and ammonium acetate. Initially, we selected the reaction of benzaldeyde, dimedone and ammonium acetate as a model reaction (Scheme 5) and the effects of catalyst loading, temperature and solvents were meticulously studied. The experimental data obtained from screening of reaction parameters are summarized in Table 3. The data indicated that using 7 mg of Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H as a sulfonic acid-functionalized titana-coated magnetic nanoparticle catalyst at 90 °C under solvent-free conditions are the optimal reaction conditions (Table 3, entry 4). It was noticed that increasing the reaction temperature and increasing the amount of nanomagnetic core-shell catalyst

did not present further improvement. Also, carrying out the model reaction in common laboratory solvents including water, EtOH, CH₃CN, EtOAc, CH₂Cl₂ and *n*-hexane did not show improved results compared to solvent-free conditions.

After determining the optimal reaction conditions, the scope and generality of the presented procedure for the synthesis of desired 1,8-dioxodecahydroacridines were established by applying a wide range of aromatic aldehydes bearing electron-releasing or electron-withdrawing groups for condensation with dimedone and ammonium acetate under the optimized reaction conditions. The data obtained, as presented in Table 4, disclose that all reactions proceeded smoothly under solvent-free conditions with high to excellent yields.

In the novel area of green chemistry, facile separation of catalyst from reaction mixture is an important issue. Magnetically recoverable catalysts find a persuasive position in green chemistry due to their merits such as high activity and selectivity and also excellent recycling and reusing capability. In a further study, in the case of both multicomponent reactions, the reusability of Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H as a sulfonic acidfunctionalized titana-coated magnetic nanoparticle catalyst was successfully investigated. At the end of the each reaction, in order to dissolve the product and unreacted starting material, hot EtOH was added to the reaction mixture and undissolved nanomagnetic catalyst was separated from it using an external magnet. In the case of

Entry	Product	Х	Y	Time (min.)	Yield (%) ^b	Melting point (°C), [found] ^{Lit.}
1	1a	Н	Н	20	80	135–137 [136–138] ^[46g]
2	1b	4-Cl	Н	20	80	125–127 [133–135] ^[46g]
3	1c	3-OH	Н	20	81	183–185 [180–180.7] ^[9b]
4	1d	4-Me	Н	30	85	113–115 [124–126] ^[17a]
5	1e	4-Cl	4-Me	35	83	142–145 [195-200] ^[46g]
6	1f	4-Cl	4-OMe	40	85	114–115 [110–112] ^[46g]
7	1g	4-Cl	4-Cl	35	82	143–145 [149–151] ^[17d]
8	1h	4-Cl	4-NO ₂	20	86	153–155 [158–161] ^[17d]
9	1i	2-Cl	4-OMe	20	85	138–140 [133–136] ^[17d]
10	1j	4-OMe	4-Me	30	80	136–138 [153–155] ^[46g]
11	1k	4-OMe	4-OMe	35	82	123–127 [135–137] ^[46g]
12	11	4-Me	4-Me	40	83	175–178 [175–177] ^[46g]
13	1m	4-Me	4-OMe	25	83	148–150 [153–155] ^[46g]
14	1n	4-Cl	2-Acetylpyridine	30	80	160–164 [173–175] ^[17e]

TABLE 2 Construction of desired molecules 1a-n in the presence of Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H^a

^aReaction conditions: arylaldehyde (1 mmol), arylketone (2 mmol) and ammonium acetate (5 mmol, 0.385 g).

^bIsolated yields.



SCHEME 5 Model reaction for screening of reaction parameters

TABLE 3 Optimizing of the reaction conditions for the synthesis of 1,8-dioxodecahydroacridines^a

Entry	Solvent	Temperature (°C)	Load of catalyst (mg)	Time (min.)	Yield (%) ^b
1	-	110	5	15	81
2	-	90	5	30	80
3	-	70	5	60	70
4	-	90	7	20	90
5	_	90	10	20	88
6 ^c	-	90	7	60	50
7^d	-	90	7	60	55
6	-	90	-	45	65
7	H ₂ O	Reflux	7	40	70
8	C ₂ H ₅ OH	Reflux	7	40	75
9	CH ₃ CN	Reflux	7	50	50
10	EtOAc	Reflux	7	50	55
11	CH_2Cl_2	Reflux	7	60	Trace
12	<i>n</i> -Hexane	Reflux	7	60	Trace

^aReaction conditions: benzaldehyde (1 mmol, 0.106 g), Dimedone (2 mmol, 0.28 g) and ammonium acetate (1 mmol, 0.077 g). ^bIsolated yields.

^cThe reaction performed using Fe_3O_4 nanoparticles as catalyst.

^dThe reaction performed using TiO₂ nanoparticles as catalyst.

2,4,6-triarylpyridine derivatives, the sequential reaction of benzaldehyde, acetophenone and ammonium acetate in 20 min (target molecule 1a) was selected as a test reaction. The recovered catalyst can be used for five continuous reaction runs. In the case of 1,8-dioxodecahydroacridine recyclability structural motifs. the of Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H was successfully investigated in the sequential reaction of 4-chlorobenzaldehyde, dimedone and ammonium acetate in 25 min (target molecule 2c). The thoroughly washed recovered catalyst can be used for five continuous reaction runs. The resulting data for both reactions are portrayed in Figure 1. For both of the described catalytic reactions turnover numbers and turnover frequencies were also calculated (see supporting information).

We propose that the final step of the mechanistic process for the construction of target molecule **1a** might proceed via anomeric-based oxidation, as depicted in Figure 2. Initially, catalyst-activated benzaldehyde is attacked by enolic form of acetophenone to form the chalcone intermediate **A**. Afterwards, a second molecule of acetophenone in enol form attacks this intermediate to afford the intermediate **B**. *In situ* generated NH₃ from dissociation of ammonium acetate $(NH_4OAc)^{[47]}$ reacts with intermediate **B** and leads to the imine intermediate **C**. In continuation, intermediate **C** is converted to **D** by a ring-closing reaction. In the next step, intermediate **D** is tautomerized to intermediate **E** which possesses a susceptible structure for the anomeric-based oxidation mechanism.^[46] In this intermediate, lone pair electron of nitrogen atom can interact with anti-bonding orbital of C—H bond (σ^*_{C-H}) in acridine moiety via resonance. This stereoelectronic structure weakens the C—H bond and facilitates H₂ release from intermediate **E** to furnish the aromatized molecule **1a**.

Figure 3 shows a plausible mechanistic pathway for the preparation of desired molecule **2a** catalysed by $Fe_3O_4@TiO_2@O_2PO_2(CH_2)_2NHSO_3H$. In the first place, activated benzaldehyde undergoes nucleophilic attack to generate the intermediate **G** through dehydration.

TABLE 4 Synthesis of target molecules 1a-q in the presence of nano magnetic catalyst 1^a

Entry	Product	R	Time (min.)	Yield (%) ^b	Melting point (°C), [found] ^{Lit.}
1	2a	Н	20	90	246–248 [258–260] ^[27j]
2	2b	2-Cl	25	85	225–227 [220–222] ^[27j]
3	2c	4-Cl	25	92	303–305 [297–299] ^[27i]
4	2 d	3-OH-4-Me	20	87	317-319 [New]
5	2e	2-OMe	15	85	298–300 [300–302] ^[27k]
6	2f	4-Br	25	93	322–324 [>300] ^[27i]
7	2g	3-NO ₂	60	81	295–298 [288–290] ^[27g]
8	2h	4-Me	35	87	318-320 [>300] ^[27i]
9	2i	3-OEt-4-OH	30	89	282–284 [New]
10	2j	3,4-(OMe) ₂	25	89	272–274 [261–263] ^[27i]
11	2k	3,4,5-(OMe) ₃	60	84	250–252 [258–261] ^[27p]
12	21	3,5-(F) ₂	90	80	301-303 [172-174] ^[27d]
13	2m	2-NO ₂	90	83	295–297 [293–295] ^[27j]
14	2n	4-CN	45	82	318–320 [324–326] ^[27g]
15	20	4-OMe	15	93	269–271 [272–274] ^[27i]
16	2p	Terephetaldehyde	20	92	270–272 [282–284] ^[28]
17	2q	Thiophen-2-carbaldehyde	35	84	313–315 [330–333] ^[271]

^aReaction conditions: aryldehyde (1 mmol), dimedone (2 mmol, 0.28 g) and ammonium acetate (1 mmol, 0.077 g),

^bIsolated yields





In the next step, dimedone in enol form attacks Knoevenagel intermediate **G** to afford intermediate **H** which id converted to imine intermediate **I** by the reaction with *in situ* generated NH_3 from NH_4OAc . Finally, a sequence of catalytic tautomerization and intramolecular nucleophilic attack furnishes the desired molecule **2a**.

Since knowledge-based development of 'anomericbased oxidation' is our main interest, similar to 2,4,6triarylpyridines, we investigated this phenomenon for the other case of 1,8-dioxodecahydroacridines. The reaction was conducted between aryl aldehydes, dimedone and ammonium acetate. In contrast to our prediction, we observed that selective production of 1,8dioxodecahydroacridines occurred and their corresponding oxidized and/or aromatized derivatives were not produced. We believe that the unpaired electron of the nitrogen atom within the 1,8-dioxodecahydroacridine structure showed a favourable resonance interaction towards electron-withdrawing carbonyl groups. This



FIGURE 2 Plausible mechanistic process for the synthesis of target molecule 1a via anomeric-based oxidation



FIGURE 3 Plausible mechanistic pathway for the construction of target molecule 2a

phenomenon does not allow the lone pair of the nitrogen atom to interact with the anti-bonding orbital of C—H bond in the 1,4-dihydropyridine moiety of 1,8-

dioxodecahydroacridine. Therefore, the non-aromatized 1,8-dioxodecahydroacridines are the preferred structures (molecules **2a–q**).

3 | CONCLUSIONS

In summary, the catalytic applicability of Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H as а sulfonic acid-functionalized titana-coated magnetic nanoparticle explored in the construction catalyst was of 2,4,6-triarylpyridines and 1,8-dioxodecahydroacridines under mild and solvent-free reaction conditions. In the case of 2,4,6-triarylpyridine derivatives, a plausible mechanism suggests an anomeric-based oxidation route to desired molecules as confirmed by experimental data. Also, the applied nanomagnetic core-shell catalyst has excellent reusability in both the investigated multicomponent reactions.

4 | EXPERIMENTAL

4.1 | General

All starting materials were obtained from Merck and applied without additional purification. The structures of known products were confirmed by comparison of their physical properties and spectral data with those of authentic samples reported in the literature. The reaction progress and purity of the prepared molecules were monitored by TLC performed with silica gel SIL G/UV 254 plates. The ¹H NMR (400 MHz) and ¹³C NMR (100 MHz) spectra were recorded with a Bruker spectrometer. Melting points were recorded with a Buchi B-545 apparatus in open capillary tubes and are uncorrected.

4.2 | General procedure for construction of magnetically recoverable solid acid catalyst Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H

The sulfonic acid-functionalized titana-coated magnetic nanoparticles, namely $Fe_3O_4@TiO_2@O_2PO_2(CH_2)_2NHSO_3H$, were prepared based on our recently reported protocol, as visualized in Scheme 3.^[46b]

4.3 | General procedure for synthesis of 2,4,6-Triarylpyridine derivatives

In a test tube containing a mixture of aromatic aldehydes (1 mmol), acetophenone derivatives (2 mmol) and ammonium acetate (5 mmol, 0.385 g), 10 mg of $Fe_3O_4@TiO_2@O_2PO_2(CH_2)_2NHSO_3H$ as a sulfonic acidfunctionalized titana-coated magnetic nanoparticle catalyst was added. The obtained reaction mixture was stirred under solvent-free conditions at 110 °C according to the appropriate times (Table 1). The progress of the reactions was followed by TLC using a mixture of *n*-hexane and EtOAc as eluent. After completion of the reactions, boiling ethanol was added to the reaction mixtures to dissolve the target products and unreacted starting materials. Then, undissolved nanomagnetic $Fe_3O_4@TiO_2@O_2PO_2(CH_2)_2NHSO_3H$ catalyst was easily separated from the reaction mixture by utilizing an external magnet, washed thoroughly with EtOH and recovered for subsequent reaction. The desired products were obtained by recrystallization from EtOH.

4.4 | General procedure for synthesis of 1,8-Dioxodecahydroacridine derivatives

In a test tube containing a mixture of aromatic aldehydes (1 mmol), dimedone (2 mmol, 0.28 g) and ammonium acetate (1mmol. 0.077 g), mg 7 of Fe₃O₄@TiO₂@O₂PO₂(CH₂)₂NHSO₃H as a sulfonic acidfunctionalized titana-coated magnetic nanoparticle catalyst was added. The resulting mixture was stirred for suitable times under optimal reaction conditions (Table 3) and the resulting data are summarized in Table 4. The reaction progress was monitored by TLC using n-hexane and EtOAc as eluent. After the reactions were completed, in order to separate the catalyst, hot EtOH was added to the reaction mixtures and desired products and unreacted starting materials were dissolved. Afterwards, undissolved nanomagnetic acidic catalyst was easily separated from the reaction mixture by utilizing an external magnet, washed with EtOH and recovered for subsequent reaction. The pure products were obtained via recrystallization from EtOH.

4.5 | Selected spectral data

3-(2,6-Diphenylpyridin-4-yl)phenol (**1c**). M.p. 183–185 °C. FT-IR (KBr, ν , cm⁻¹): 3034, 1606, 1596, 1543, 1493, 1198, 764. ¹H NMR (400 MHz, DMSO, δ , ppm): 9.73 (s, 1H, OH), 8.37 (d, 4H, J = 8 Hz, aromatic), 8.17 (s, 2H, aromatic), 7.61 (t, 4H, J = 8 Hz, aromatic), 7.56–7.48 (m, 3H, aromatic), 7.44–7.42 (m, 2H, aromatic), 6.98 (d, 1H, J = 8 Hz, aromatic). ¹³C NMR (101 MHz, DMSO, δ , ppm): 158.0, 156.4, 149.8, 139.2, 138.8, 130.1, 129.2, 128.7, 126.9, 118.0, 116.5, 116.2, 114.0.

4-(4-Chlorophenyl)-2,6-di-*p*-tolylpyridine (**1e**). M.p. 142–145 °C. FT-IR (KBr, ν , cm⁻¹): 3028, 1655, 1599, 1491, 1407, 1224, 774. ¹H NMR (400 MHz, DMSO, δ , ppm) 8.14 (d, 2H, J = 8 Hz, aromatic), 8.05–7.99 (m, 4H, aromatic), 7.78 (d, 2H, J = 16 Hz,aromatic), 7.69 (d, 3H, J = 8 Hz,aromatic), 7.69 (d, 3H, J = 8 Hz,aromatic), 7.45 (d, 3H, J = 8 Hz,aromatic), 2.47 (s, 6H, Me). ¹³C NMR (101 MHz, DMSO, δ , ppm): 188.5, 143.7, 142.1, 135.0, 134.9, 133.7, 130.5, 129.4, 128.9, 128.7, 123.2, 21.2.

4-(4-Chlorophenyl)-2,6-bis(4-methoxyphenyl)pyridine (**1f**). M.p. 114–115 °C. FT-IR (KBr, ν , cm⁻¹): 2962, 1656, 1605, 1511, 1492, 1177, 817. ¹H NMR (400 MHz, DMSO, δ , ppm): 8.23 (d, 2H, J = 8 Hz, aromatic), 8.05–7.97 (m, 4H, aromatic), 7.75 (d, 2H, J = 8 Hz, aromatic), 7.58 (d, 3H, J = 8 Hz, aromatic), 7.15 (d, 3H, J = 8 Hz, aromatic), 3.93 (s, 6H, OMe). ¹³C NMR (101 MHz, DMSO, δ , ppm): 187.2, 163.3, 141.6, 134.8, 133.8, 131.0, 130.4, 130.3, 128.9, 122.8, 114.0, 55.6.

4-(4-Methoxyphenyl)-2,6-di-*p*-tolylpyridine (**1**j). M.p. 183–185 °C. FT-IR (KBr, ν , cm⁻¹): 3061, 1609, 1597, 1509, 1424, 1180, 820. ¹H NMR (400 MHz, DMSO, δ , ppm): 8.27 (d, 4H, J = 8 Hz, aromatic), 8.13 (s, 2H, aromatic), 8.06 (d, 2H, J = 8 Hz, aromatic), 7.40 (d, 4H, J = 8 Hz, aromatic), 7.40 (d, 4H, J = 8 Hz, aromatic), 7.16 (d, 2H, J = 8 Hz, aromatic), 3.90 (s, 3H, OMe), 2.44 (s, 6H, Me). ¹³C NMR (101 MHz, DMSO, δ , ppm): 160.3, 156.3, 148.9, 138.6, 136.2, 130.7, 129.9, 129.3, 128.6, 126.8, 115.2, 114.4, 55.3, 20.9.

2,4,6-Tris(4-methoxyphenyl)pyridine (**1k**). M.p. 123– 127 °C. FT-IR (KBr, ν , cm⁻¹): 3003, 2836, 1608, 1583, 1509, 1427, 1174, 822. ¹H NMR (400 MHz, DMSO, δ , ppm): 8.33 (d, 4H, J = 8 Hz, aromatic), 8.07–8.04 (m, 4H, aromatic), 7.16 (t, 6H, J = 16 Hz, aromatic), 3.91 (s, 9H, OMe). ¹³C NMR (101 MHz, DMSO, δ , ppm): 160.2, 155.9, 148.8, 131.5, 130.1, 128.5, 128.2, 114.4, 114.3, 114.0, 55.2.

4'-(4-Chlorophenyl)-2,2':6',2"'-terpyridine (**1n**). M.p. 160–164 °C. FT-IR (KBr, ν , cm⁻¹): 3058, 1602, 1583, 1549, 1471, 1091, 786. ¹H NMR (400 MHz, DMSO, δ , ppm): 9.09 (s, 1H, aromatic), 8.81–8.63 (m, 4H, aromatic), 8.38 (s, 3H, aromatic), 8.02 (s, 2H, aromatic), 7.62–7.53 (m, 4H, aromatic). ¹³C NMR (101 MHz, DMSO, δ , ppm): 155.9, 155.2, 155.0, 153.4, 149.9, 149.3, 148.1, 137.6, 137.4, 137.2, 134.2, 129.3, 128.8, 128.6, 124.5, 124.5, 121.5, 120.9, 120.8, 117.8, 117.3, 116.5.

9-(3-Hydroxy-4-methylphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8-(2*H*,5*H*)-dione (2d). M.p. 317–319 °C. FT-IR (KBr, ν , cm⁻¹): 3538, 2958, 1650, 1607, 1489, 1367, 1143, 759. ¹H NMR (400 MHz, DMSO, δ , ppm): 8.99 (s, 1H, NH), 8.38 (s, 1H, OH), 6.43–6.29 (m, 3H, aromatic), 4.45 (s, 1H, CH), 3.12 (s, 3H, Me), 2.25–1.77 (m, 8H, CH₂), 0.76 (s, 6H, Me), 0.66 (s, 6H, Me). ¹³C NMR (101 MHz, DMSO, δ , ppm): 194.33, 148.88, 145.53, 140.12, 118.05, 115.47, 111.69, 111.47, 55.50, 50.31, 32.09, 31.82, 29.10, 26.57.

9-(2-Methoxyphenyl)-3,3,6,6,tetramethyl-3,4,6,7,9,10hexahydroacridine-1,8-(2*H*,5*H*)-dione (**2e**). M.p. 298– 300 °C. FT-IR (KBr, ν , cm⁻¹): 3285, 2955, 1645, 1605, 1489, 1224, 1143, 747. ¹H NMR (400 MHz, DMSO, δ , ppm): 8.97 (s, 1H, NH), 6.94–6.56 (m, 4H, aromatic), 4.69 (s, 1H, CH), 3.43 (s, 3H, OMe), 2.26–1.66 (m, 8H, CH₂), 0.76 (s, 6H, Me), 0.58 (s, 6H, Me). ¹³C NMR (101 MHz, DMSO, δ , ppm): 194.02, 157.57, 149.59, 133.81, 131.40, 126.65, 119.11, 110.76, 110.02, 54.98, 50.37, 31.95, 31.38, 29.31, 25.85.

9-(3-Ethoxy-4-hydroxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8-(2*H*,5*H*)-dione (**2i**). M. p. 282–284 °C. FT-IR (KBr, ν , cm⁻¹): 3276, 2966, 1646, 1620, 1478, 1365, 1221, 1144. ¹H NMR (400 MHz, DMSO, δ , ppm): 9.32 (s, 1H, NH), 8.60 (s, 1H, OH), 6.80–6.64 (m, 3H, aromatic), 4.82 (s, 1H, CH), 4.00 (br s, 2H, OCH₂), 2.61–2.12 (m, 8H, CH₂), 1.39 (br s, 3H, Me), 1.12 (s, 6H, Me), 1.00 (s, 6H, Me). ¹³C NMR (101 MHz, DMSO, δ , ppm): 194.43, 148.93, 145.72, 144.67, 138.40, 63.79, 50.30, 32.08, 31.81, 29.14, 26.34, 14.75.

9-(3,5-Difluorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8-(2*H*,5*H*)-dione (**2l**). M. p. 301–303 °C. FT-IR (KBr, ν , cm⁻¹): 3277, 2961, 1646, 1619, 1488, 1365, 1222, 1144, 987. ¹H NMR (400 MHz, DMSO, δ , ppm): 9.45 (s, 1H, NH), 6.91–6.79 (m, 3H, aromatic), 4.89 (s, 1H, CH), 2.45–2.13 (m, 8H, CH₂), 1.00 (s, 6H, Me), 0.92 (s, 6H, Me). ¹³C NMR (101 MHz, DMSO, δ , ppm): 194.45, 163.13, 160.71, 151.39, 150.01, 110.50,

110.27, 101.07, 50.05, 33.14, 32.12, 28.88, 26.44. 9-(4-Methoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10hexahydroacridine-1,8-(2*H*,5*H*)-dione (**2o**). M.p. 269–271 °C. FT-IR (KBr, ν, cm⁻¹): 3279, 2958, 1644, 1634, 1606, 1483, 1368, 1145, 834. ¹H NMR (400 MHz, DMSO, δ , ppm): 9.25 (s, 1H, NH), 7.06–6.73 (m, 4H, aromatic), 4.77 (s, 1H, CH), 3.67 (s, 3H, OMe), 2.19–2.02 (m, 8H, CH₂), 1.00 (s, 6H, Me), 0.90 (s, 6H, Me). ¹³C NMR (101 MHz, DMSO, δ , ppm): 194.34, 157.06, 149.00, 139.49, 128.49, 112.89, 111.67, 54.79, 50.25, 32.10, 31.86, 29.09, 26.47.

3,3,6,6-Tetramethyl-9-(thiophen-2-yl)-3,4,6,7,9,10hexahydroacridine-1,8-(2*H*,5*H*)-dione (**2q**). M.p. 313–315 °C. FT-IR (KBr, ν , cm⁻¹): 3277, 2956, 1638, 1626, 1605, 1482, 1371, 1217, 716. ¹H NMR (400 MHz, DMSO, δ , ppm): 9.25 (s, 1H, NH), 6.95–6.55 (m, 3H, aromatic), 4.95 (s, 1H, CH), 2.01–2.28 (m, 8H, CH₂), 0.76 (s, 12H, Me). ¹³C NMR (101 MHz, DMSO, δ , ppm): 194.3, 192.4, 149.6, 126.2, 123.0, 122.8, 110.8, 50.2, 32.7, 32.42, 28.78, 27.1, 26.5.

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